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FINAL REPORT,

(NASA Contract No. NASw-734)

INVESTIGATION OF ADHESION AND COHESION OF METALS IN ULTRAHIGH VACUUM

March 1, 1963 -- November 1, 1963

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ABSTRACT

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Apparatus was developed for the measurement of the force necessary to separate small specimens of metal cleaned and joined in ultrahigh vacuum. Equipment previously used under Contract NASr-48 for repeatedly fracturing and rejoining a single specimen was fitted with indexing specimen holders so that eight cohesion tests could be made with a single pumpdown.

The fracture-rejoin technique eliminates the surface cleaning problem and permits acquisition of data characteristic of the metal itself; it is not applicable to dissimilar metal pairs. Both similar and dissimilar combinations of the following metals at two hardness levels were studied -- copper, copper-beryllium alloy, 1018 steel, 4140 steel, 440C steel and titanium. The cleaning method used was wire brushing.

Both flat faced and chisel edged specimens were used; the rectangular faces or chisel edges being crossed. All tests were at room temperature and at pressures between 10^{-8} and 10^{-9} torr. Three runs (24 tests) were made with flat specimens and one run (8 tests) with chisel edge specimens. Cohesion occurred only between flat faced soft copper specimens wire brushed in vacuum. The cohesive force varied from 8 to 120 lbs. after a compressive force of 2000 lbs. and appeared to depend primarily on the thoroughness of wire brushing. The need for further tests is emphasized.

AUTHOR

FOREWORD

This is the summary report of work performed in the Research Division of National Research Corporation, under Contract No. NASw-734 for NASA Headquarters, and covers the period March 1, 1963 to November 1, 1963.

The general object of the work is to obtain additional information as to the conditions under which metals and alloys of engineering importance for space applications will adhere to one another with sufficient tenacity to hinder the relative motion or subsequent separation of components of mechanical and electrical devices, used in space exploration. Such devices include bearings, solenoids, valves, slip rings, mating flanges, conical rendezvous mating surfaces and similar components.

Major contributors to this program were, Dr. Frank C. Benner, Program Director; Mr. John L. Ham, Research Associate; Mr. Charles Mariano, Physicist, all of the National Research Corporation and Dr. George S. Reichenbach, Associate Professor and Consultant, Massachusetts Institute of Technology.

INTRODUCTION

Research on the tendency of clean metals to stick together in high vacuum has been carried on at National Research Corporation for the past three years (1,2,3) under NASA Contracts NASr-48 and NASw-734.

The general objective has been to obtain additional information as to the conditions under which metals and alloys of engineering importance for space applications will adhere to one another with sufficient tenacity to hinder the relative motion or subsequent separation of components of mechanical and electrical devices used in space exploration. Such devices include bearings, solenoids, valves, slip rings, mating flanges, conical rendezvous mating surfaces, etc.

Under Contract NASr-48 techniques were developed for evaluating the cohesion of metals at various temperatures by repeatedly fracturing and rejoing notched tensile specimens in ultrahigh vacuum. Two types of apparatus were used: 1) a differential expansion device and 2) a screw drive device. The latter was found to be the better. The maximum cohesion obtained at room temperature was about 65% for copper, 19% for 1018 steel, and zero percent for hardened 52100 steel. Time in contact appears to be an important factor for copper at 200°C and above. Both 1018 steel and 52100 steel were "self-cleaning" at 500°C; the former showing repeated readings near 100% cohesion, and the latter increasing in percent cohesion with each successive break at 500°C.

However, hardness turned out to be an important variable with respect to cohesion, and it was found that at room temperature copper or mild steel when repeatedly fractured and rejoined in vacuum exhibited less and less cohesion even at ambient pressures believed to be too low to permit significant contamination in the time available. These initially soft metals were work hardened by this treatment and the successive reductions in cohesion are ascribed to this hardening. No cohesion could be measured on initially hard heat treated 52100 steel at room temperature when tested in a similar manner.

Although there are many other important variables such as temperature, time in contact, degree of deformation in compression, sliding, etc., it was considered most important, from a practical standpoint, to first assess the tendency of various commonly used alloys to stick together at room temperature without sliding or severe deformation. It was also considered advisable to determine the behavior of clean surfaces before attempting to study the complex variable of degree of contamination. Exposure to vacuum alone at room temperature can remove only certain physisorbed gases (4) and only mild heating in vacuum can be tolerated when the effect of work hardening or even of hardening by heat treatment is to be evaluated.

Therefore, in the present contract (NASw-734) the equipment was modified to permit contacting a number of specimens successively without breaking the vacuum. Means for cleaning the surfaces of these specimens while under vacuum were also provided. The first method successfully used was wire brushing. Some development of a second method, ion bombardment, was also begun and this work is described in Appendix A.

The materials studied for cohesion/adhesion properties were soft and hard specimens of each of the following: O.F.H.C. copper, 1018 steel, 440C stainless steel, 4140 steel, Cu-Be alloy, commercially pure titanium, and coin silver.

APPARATUS

The apparatus consisted of a stainless steel vacuum chamber with the accessories necessary to join and separate small metal specimens in ultrahigh vacuum and to measure the forces involved. The major components were developed and used in previous programs.

Fig. 1 shows the loading and force measuring devices which communicate with the inside apparatus through a flexible metal bellows. Beneath the dome in Fig. 1 hangs the apparatus shown in Figs. 2 and 3. Sixteen specimens (eight pairs) can be mounted on the wheels shown in Figs. 2 and 3, and indexed to bring different material combinations together or to expose a given surface for cleaning.

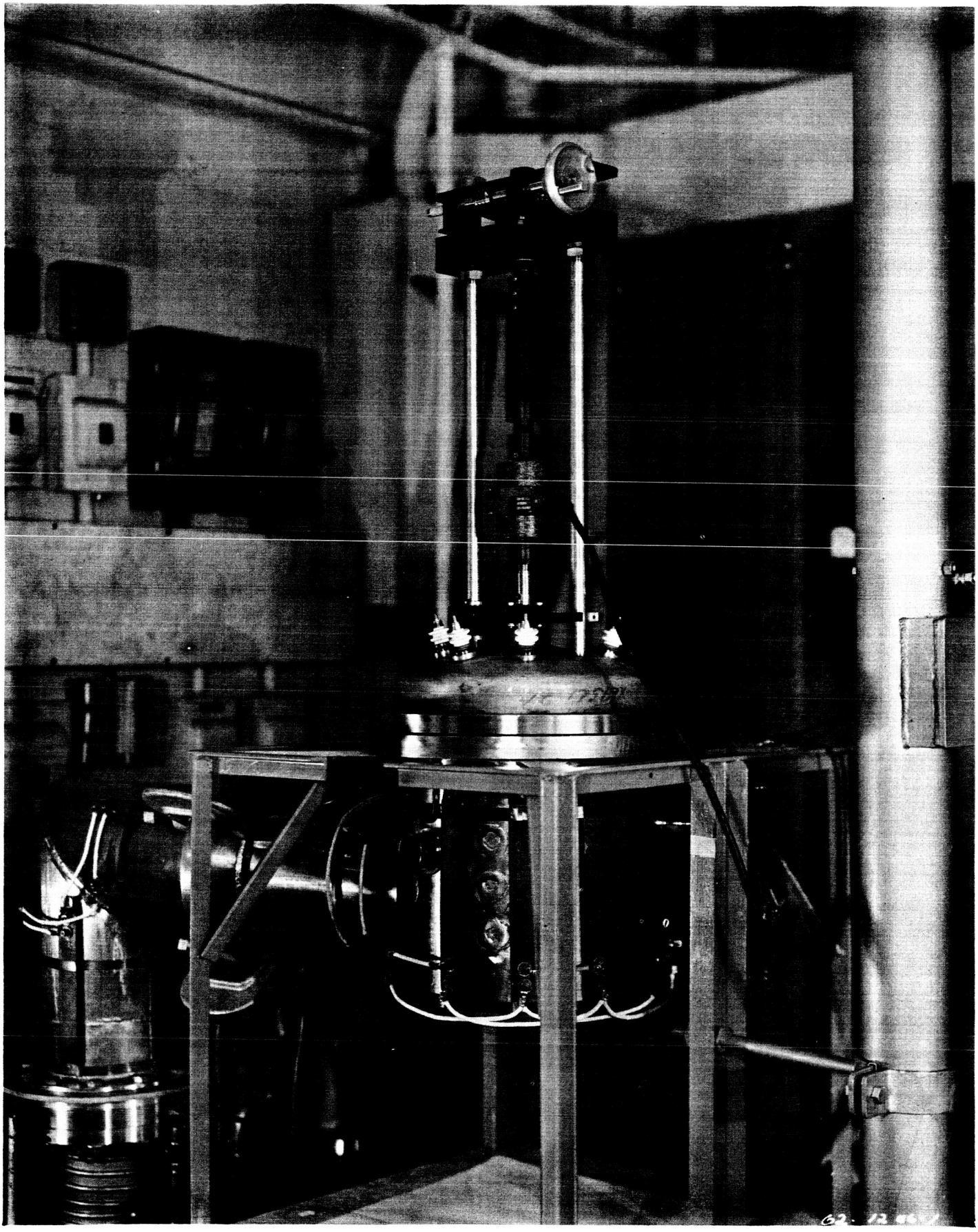

Figure 1 - Screw Drive Cohesion Testing Apparatus (outside)

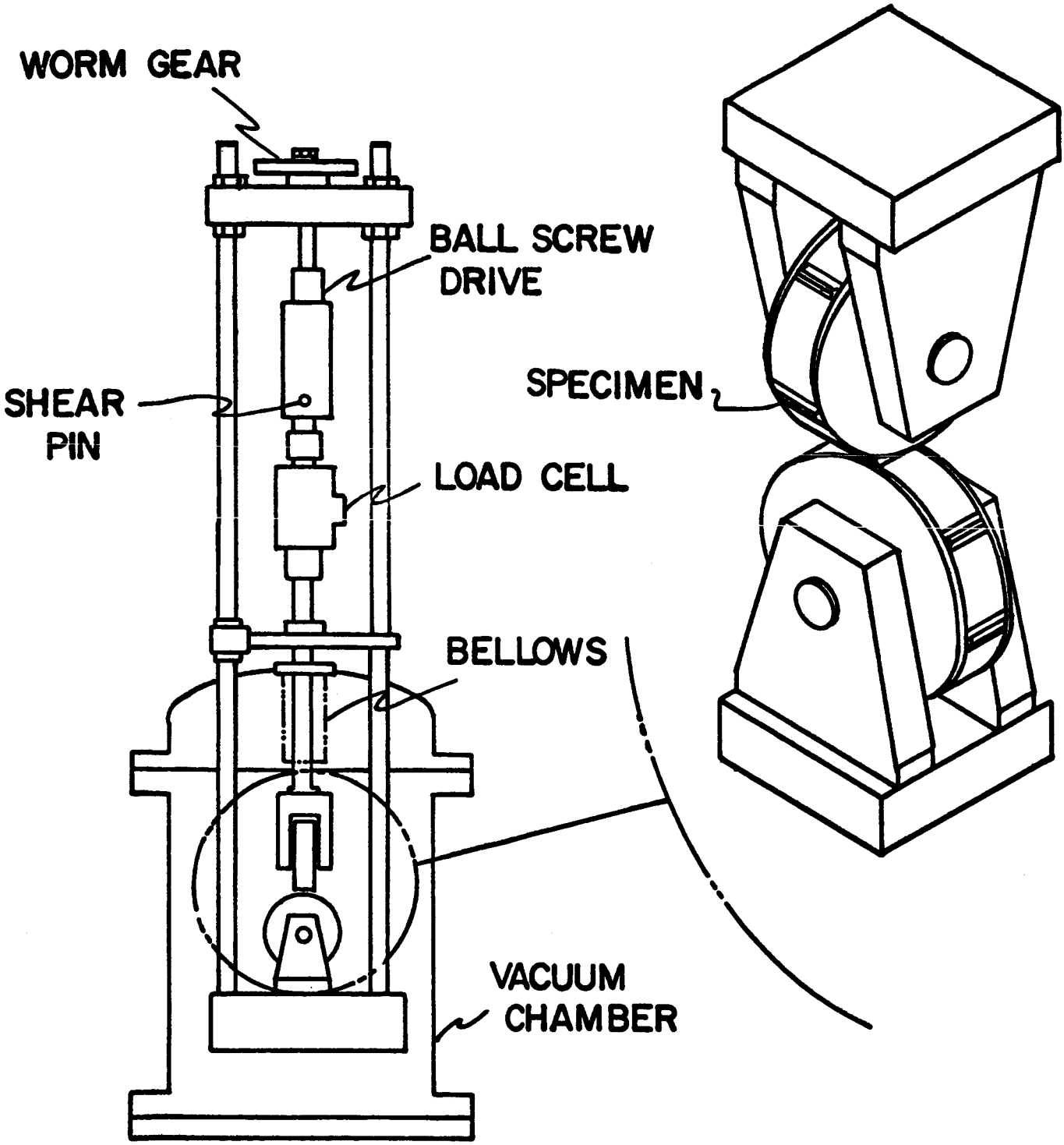

FIGURE 2 - COHESION TEST APPARATUS

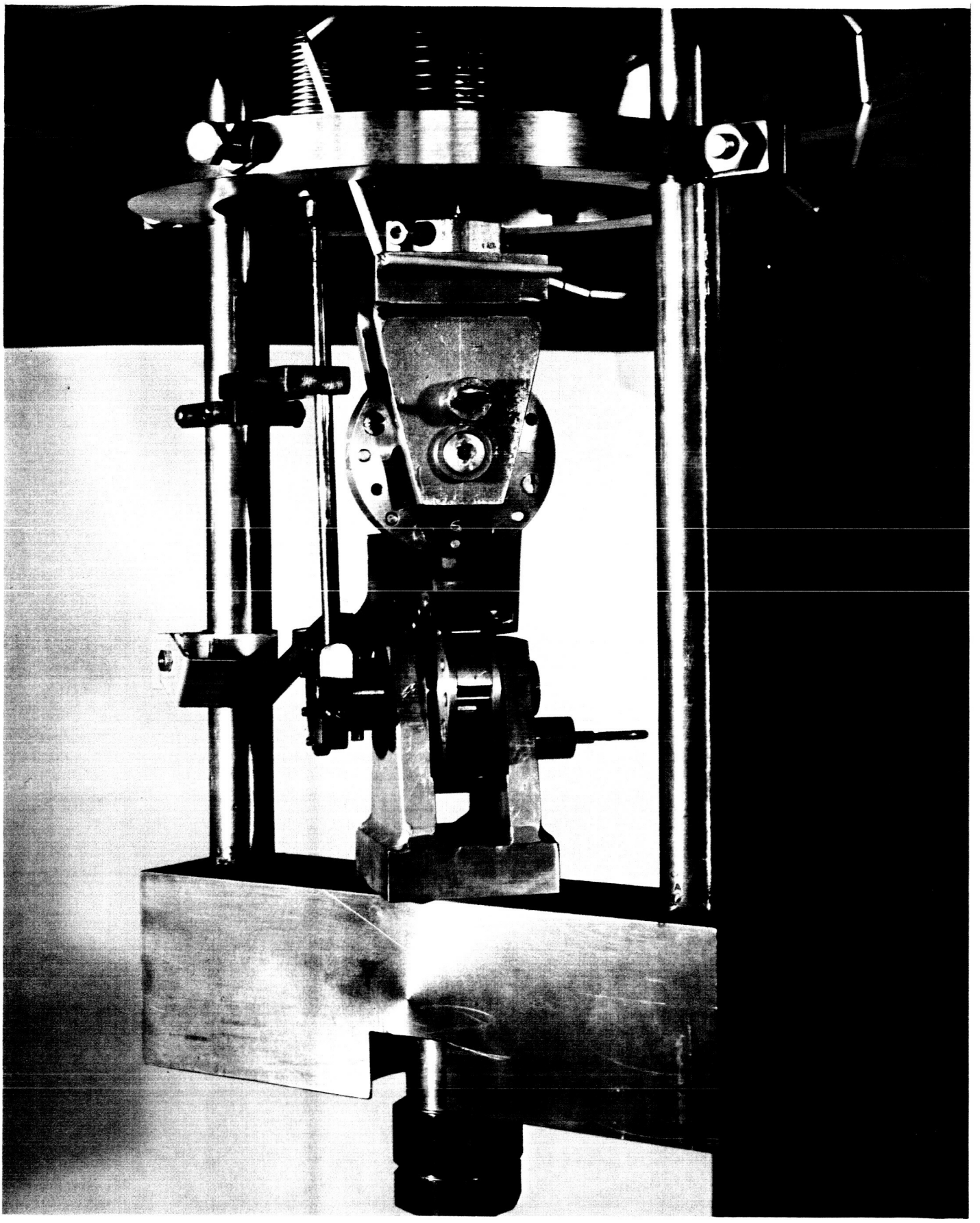

Figure 3 - Indexing Sample Holder

A twelve-inch long spool piece is now located between the dome and the bowl of Fig. 1 (but not shown in figure).

The spool piece accommodates windows, for projecting an image of the specimens on a screen, and a bellows manipulator for pushing and pulling a wire brush or other abrasive tools in and out between specimens just before joining.

The entire assembly is mounted on a standard NRC ultrahigh vacuum pumping system with a 10-inch diffusion pump (HS10-4200) and standard NRC Chevron type liquid nitrogen trap. Concentric "O"-Rings cooled by a circulating refrigerant are used at the joints between the large flanges.

SPECIMENS

The type of specimen used is shown in Fig. 4. Eight of these fit into each wheel of Figs. 2 and 3. They are held by hardened pins which permit either specimen of a pair to adjust itself parallel to the other. This minimizes the possibility of misalignment during either compression or tension.

The face widths of the specimens are adjusted to permit a few thousandths of an inch face depression with compressive loads of 2000 to 3000 lbs. Some specimens were machined to a chisel edge (zero face width). The surfaces are prepared by mounting three specimens at a time in a special jig and abrading with successively finer grades of alundum or emery paper. The three specimens form a triangle in the polishing jig and a flat plate is used under the abrasive paper. This insures specimen flatness. A slightly different type of polishing

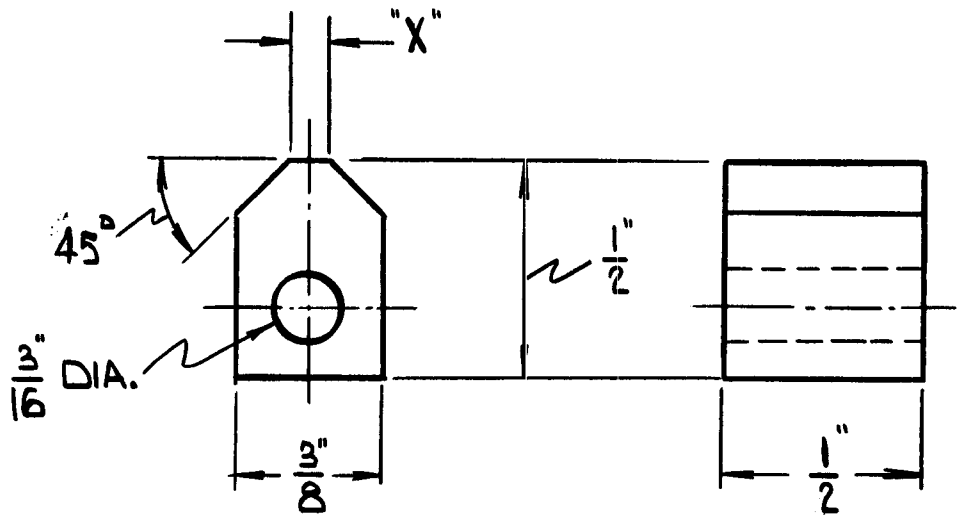

Figure 4 - Adhesion Specimen

jig was used for preparing the two flat faces of 90° chisel type specimens. Just before mounting in the test wheel, each specimen is given one rub on dry No. 000 paper.

The hard specimens of O.F.H.C copper and 1018 steel were made directly from cold drawn 3/4-inch diameter bar stock. The soft copper and soft 1018 steel specimens were made from the same stock after annealing. The 3/4-inch diameter titanium stock could be obtained only in the condition referred to commercially as "annealed". Actually this condition is quite hard so that by annealing a portion of such a bar both hard and soft materials were obtained. The Cu-Be alloy (No. 25) was machined to size from 3/4-inch diameter bar stock in the solution treated condition and some of the specimens hardened by the standard aging treatment prior to finishing on the No. 000 paper and beveling by grinding. The 440C stainless steel and the 4140 steel specimen were machined to size from soft bar stock except for .010 inches to be removed from the face of the hard ones by surface grinding to remove decarburized metal prior to finishing on the 000 paper. The 4140 steel was drawn to about 50 Rockwell C. The 440C steel and Cu-Be was left at maximum hardness.

The specimens prepared are described in Table I. Some of these specimens were re-machined to a chisel point.

Table II shows the sample combinations selected for testing.

TABLE I

Number of Specimens Required

of each Material and Face Width

Material	Face Width (inches)	Number Required	Material	Face Width (inches)	Number Required
Soft Copper	.25	35	Soft Cu-Be	.25	2
				.15	5
Hard Copper	.25	2			
	.18	4	Hard Cu-Be	.25	2
				.15	2
Soft 1018	.25	2		.10	4
	.18	16			
			Soft Ti	.25	2
				.22	5
Hard 1018	.25	2			
	.13	5	Hard Ti	.25	2
				.22	2
Soft 440C	.25	2		.14	4
	.13	5			
			Soft		
Hard 440C	.25	2	Coin Silver	-	2
	.13	2		-	5
	.08	4			
			Hard		
Soft 4140	.25	2	Coin Silver	-	2
	.10	5		-	5
Hard 4140	.25	2			
	.10	5			

TABLE II

SAMPLE COMBINATIONS FOR 1963 COHESION TESTS

		Copper		1018		440C		4140		Cu-Be		Coin Ag		Ti	
		S	H	S	H	S	H	S	H	S	H	S	H	S	H
Copper	S	*	x	x	x	x	x	x	x	x	x	x	x	x	x
	H		x												
1018 Steel	S			*	x										
	H				x										
440C Steel	S					x	x								
	H						x								
4140 Steel	S							x	x						
	H								x						
Cu-Be	S									x	x				
	H										x				
Coin Ag	S											x	x		
	H												x		
Ti	S													x	x
	H														x

* Multiple tests for comparison of wire brushing and ion bombardment.

x Single tests with either wire brushing or ion bombardment.

S Soft H Hard

EXPERIMENTAL PROCEDURE

Four test runs were made. In each case all sixteen stations of the indexing wheels were filled with specimens selected from Table II and prepared as already described. The system was baked out under vacuum before each run, using quartz lamps mounted inside the vacuum chamber. The specimen holders reached 250 to 280°C during bake out. In most cases the specimens were wire brushed just before joining by means of a very small two-sided stainless steel brush activated by a rod through a flexible bellows. The specimens were indexed to face one another and were close enough together to cause some distortion of the brush as it passed between them. However, due to mechanical difficulties, the specimens were brushed as thoroughly as was desired in only one of the runs. No attempt was made to brush the chisel edge specimens.

Forces ranging from 1400 to 3000 pounds were used for joining and force was generally applied for 60 seconds. It usually required about 30 seconds to get the specimens together after brushing. During this time the pressure was between 10^{-8} and 10^{-9} torr.

After testing, the specimens were examined under the microscope and the amount of "deformation" (depth of impressions) was measured.

Exceptions to these general procedures will be brought out in the following section.

EXPERIMENTAL RESULTS

RUN NO. 1

Four soft copper and four soft 1018 specimens were mounted in each wheel and the apparatus was pumped down and baked out at 250°C. Pressure after cooling was 2×10^{-8} torr. Although this was not quite as low as expected, it was decided to proceed with the tests in order to uncover any other possible difficulties. Mechanically the apparatus worked very well except for the wire brushing. Compressive loads to 3000 pounds were used and the indexing and load measuring devices worked well. However, brushing by lateral motion of the bellows proved difficult. (The brush was later relocated to permit push-pull brushing.)

In these tests it took about a minute to get the specimens together after brushing. This was partly due to inexperience with the apparatus and partly to an attempt to measure the exact amount of compressive deformation by means of a light beam, a mirror, a projection lens and a ground glass screen. For the small amounts of deformation desired this is quite difficult. Pressure between cleaning and joining varied from 8×10^{-9} to 2×10^{-8} torr. Force was applied for one minute except for one ten-minute test on the steel.

The results of Run No. 1 are given in Table III. It required only 32 pounds to pull the first pair of copper samples apart after one minute in contact at 1400 pounds (20,300 psi) during which one face was indented 2.7 mils and the other 2.2 mils. In the second test on copper, 1900 pounds (24,400 psi) was applied with considerable

TABLE III

ADHESION RUN NO. 1

P= 8 x 10^{-9} to 2 x 10^{-8} torr. Time in Contact = 60 seconds. 280°C Bake Out.

Test No.	Material		Comp. Force (lbs)	Contact Area (in^2)	Comp. Stress (1000 psi)	Deformation (Mils)		Cohesion Force (lbs.)	Stress (psi)
	Top	Bottom				Top	Bottom		
1	Soft Cu	Soft Cu	1400	.0694	20.2	2.7	2.2	32	461
2	Soft Cu	Soft Cu	1900	.0777	24.4	5.2	3.9	24	310
3	Soft Cu	Soft Cu	2600	.0677	38.4	8.1	9.3	(pin sheared)	-
4	Soft Cu	Soft Cu	1900	.0656	29.0	4.7	5.2	0*	0
5	Soft 1018	Soft 1018	3000	.0342	87.7	1.6	1.4	0	0
6	Soft 1018	Soft 1018	3000	.0364	82.4	0.3	0.1	0	0
7	Soft 1018	Soft 1018	3000	.0361	83.1	1.5	1.4	0	0
8	Soft 1018	Soft 1018	--	--	-	-	-	(no test)	-

* Not Wire Brushed

resulting deformation but it required only 24 pounds to pull the samples apart. In the third test a safety pin was sheared off at 2600 lbs. and no data obtained. In the fourth test, copper samples were pushed together with 1900 pounds force (29,000 psi) without wire brushing. No sticking occurred. (Possibly more of the surface was effectively brushed in test one than in test two, though this was not evident on visual inspection.)

Three of the soft steel pairs were joined at 3000 pounds (83,000 to 88,000 psi) after wire brushing but no cohesion occurred even though some compressive deformation (0.1 to 1.6 mils) was observed.

Examination of the samples after testing showed plainly that only portions of the mating surfaces were effectively brushed. Each brush scratch on the copper was bright red. Apparently, the bright red type of oxide, or oxide of a thickness such that it appears red, forms in vacuum at room temperature. On standing in air the color gradually changed to the color usually associated with copper.

RUN NO. 2

Eight copper specimens and eight soft 1018 steel specimens were polished on 000 aloxite abrasive paper as usual. With four soft copper specimen pairs and four 1018 specimen pairs in place, the apparatus was pumped down and baked out at 280°C. The maximum pressure (4.4×10^{-6} torr) occurred at 225°C. Final pressure at 282°C was 1.5×10^{-6} torr. Final pressure at room temperature was 1.0×10^{-9} torr. Each pair of specimens (except one) was then wire brushed and pushed together

until it deformed about .010 inches (.005 inches on each side). One of the copper pairs was pushed together without wire brushing and one pair was allowed to stand for 10 minutes at 1.4×10^{-9} torr after wire brushing before being pushed together. One steel pair was held in compression for 11 minutes.

The results and exact conditions of the tests are recorded in Table IV.

All of the soft copper specimens except the one not wire brushed stuck together with measurable force, but none of the steel specimens stuck together with measurable force. The largest cohesive force obtained was 120 lbs. (1790 psi) which represents a cohesion co-efficient of 120/2000 or 0.06. In this test the specimens were intentionally left apart for 10 minutes at 1.4×10^{-9} torr. However, this represents an exposure of only 1.4×10^{-8} torr min. which is sufficient for only a small fraction of a monolayer to form. Therefore, it was assumed that the cleanliness of all three of the stuck copper pairs was the same.

From Test No. 3 and from the previous run, the brushing is known to be effective either because of the roughening effect or the cleaning effect or both, and it is believed that the thoroughness of the brushing was about the same for all the specimens in this run. Therefore, since the three cohesion values obtained correlate with the applied stress and strain it is concluded that the observed differences in cohesive force reflect these small inadvertant differences in contact area and load. The specimen not wire brushed gave zero cohesion even though the stress was 29,900 psi and the deformation was 11.8 mils. The fact that the steel did not bond even in 11 minutes after 9.1 mils total deformation indicates that either hardness or degree of roughening or both are indeed important

TABLE IV

ADHESION RUN NO. 2

$P = 1 \times 10^{-9}$ to 2×10^{-9} torr. Time in Contact = 60 sec. (except as noted) 250°C Bake Out

Test	Material		Comp. Force (lbs.)	Contact Area (in^2)	Comp. Stress (1000 psi)	Deformation (mils)		Cohesion		Time Apart After Brushing (sec)
	Top	Bottom				Top	Bottom	Force (lbs.)	Stress (Psi)	
1	Soft Cu	Soft Cu	2000	.0702	28.5	3.6	4.5	60	854	70
2	Soft Cu	Soft Cu	1950	.0668	29.2	4.7	4.5	100	1498	30
3	Soft Cu	Soft Cu	2000	.0668	29.9	6.1	5.7	0*	0	*
4	Soft Cu	Soft Cu	2000	.0635	31.5	5.3	6.4	120	1790	600
5	Soft 1018	Soft 1018	2800	.0202	138.5	4.6	5.0	+	--	30
6	Soft 1018	Soft 1018	2300	.0216	106.5	2.3	2.3	0	0	30
7	Soft 1018	Soft 1018	2400	.0172	139.5	4.9	4.2	0	0	30++
8	Soft 1018	Soft 1018	-	-	-	-	-	-	-	-

* - No Brushing

\+ - Pin Sheared

++ - Held in Contact for 660 sec.

factors. The brushing causes less roughening of the steel than of the copper.

RUN NO. 3

In Run No. 3 (Table V) flat specimens of the usual type were used in an attempt to determine whether soft copper would stick to other metals as tenaciously as to itself after wire brushing in vacuum. Unfortunately the wire brushing device failed during this series and it was discovered on subsequent examination that only part of the surface of some specimens had actually been brushed. The estimated percentages of contact area actually brushed are given in Table V for each specimen. Adhesion occurred only between soft copper and itself and even in this case the cohesive force was only half that previously measured for well brushed specimens. As the table indicates, none of the metals other than copper was brushed well enough to determine whether it might stick to soft copper if clean.

As shown by Test No. 1 of Table V, no cohesion occurs between soft copper and soft copper if the wire brushing is done in air instead of in the vacuum even though the brushing is very thorough. Therefore, the cleaning effect of the brushing must be more important than the roughening effect, and severe cohesion, possibly equivalent to that observed in the previous program between freshly fractured surfaces, remains a possibility. By comparison with some other cleaning method such as ion bombardment or grinding or machining in vacuum, this question may be resolved. We cannot be sure how thorough the wire brush cleaning is even when all the surface appears to have been scratched by the brush.

TABLE V

ADHESION RUN NO. 3

P = 5 x 10^{-9} to 7 x 10^{-9} torr. Time in Contact = 60 Seconds (250°C Bake Out)

Test	Material		Comp. Force	Cont. Area	Comp. Stress 1000	Deformation (mils)		Cohesion		Est. Percent++ of Contact Area Effectively Brushed	
	Top	Bottom	(lbs.)	(in^2)	(Psi)	Top	Bottom	Force (lbs.)	Stress (Psi)	Top	Bottom
1	Soft Cu	Soft Cu	2400	.0688	34.9	7.0	6.3	0+	0	100(in air)	100
2	Soft Cu	Soft Cu	2200	.0701	31.5	7.0	5.6	8	114	90	10
3	Soft Cu*	Soft Cu	2400	.0655	36.6	5.2	5.4	60	915	80	60
4	Soft 1018	Soft Cu	2400	.0700	34.3	0	7.2	0	0	2	90
5	Soft 440C	Soft Cu	2400	.0675	35.5	0	6.5	0	0	1	70
6	Soft 4140	Soft Cu	2400	.0688	34.9	0	5.0	0	0	1	80
7	Soft Cu-Be Alloy	Soft Cu	2400	.0662	36.3	0	6.0	0	0	5	90
8	Soft Titanium	Soft Cu	2400	.0663	36.4	0	6.5	0	0	2	90

*This sample was initially hard but was annealed during bake out.

++ Subsequent examination of the specimens showed that, except for Tests No. 1 and No. 3, large portions of the contact areas remained unscratched by the brush.

+ Joined and parted twice. No force measurements made the first time due to electrical difficulty.

RUN NO. 4

In the fourth adhesion test run (See Table VI) a new kind of specimen was used. The usual 45° beveled edges previously used to define the width of the flat face were widened till they met at the center forming a 90° chisel edge (zero face width). Thus a test consisted of pushing crossed chisel edges together at right angles to one another. This was done in order to provide a large amount of deformation with the force available, i.e. to simulate severe roughness or mismatch. Specimen pairs of both equal and unequal hardness were represented as shown by the Table. For equal hardnesses the deformation was equal on each side and little or no sliding occurred. For unequal hardnesses all of the deformation occurred in the softer specimen and severe sliding must have occurred. No wire brushing was attempted. In spite of the large amounts of deformation and sliding no measurable adhesion occurred. However, the type of deformation applied did not greatly increase surface area,i.e. did not drastically stretch the oxide film.

This was an unexpected result, and must mean that the oxide layer on copper can withstand some stretching and sliding without rupture. It also means that deformation and sliding, though possibly necessary, are not always sufficient conditions for adhesion. It is believed that no mobile adsorbed films were present at 8×10^{-9} torr after the 250°C bake out.

TABLE VI

ADHESION RUN NO. 4

(Crossed 90° Chisel Edges)

$P = 7 \times 10^{-9}$ to 8×10^{-9} torr. Time in Contact = 60 Secs. (250°C BakeOut)

Test	Material		Comp. Force (lbs.)	Deformation* (mils)		Cohesive Force (lbs.)
	Top	Bottom		Top	Bottom	
1	Soft Cu	Soft Cu	2400	68	68	0
2	Soft 440C	Soft Cu	2400	0	104	0
3	Soft 4140	Soft Cu	2400	0	109	0
4	Soft Cu-Be	Soft Cu	2400	0	115	0
5	Soft Ti	Soft Cu	2400	0	108	0
6	Soft 1018	Soft 1018	2400	32	32	0
7	Soft Ti	Soft Ti	2400	20	20	0
8	Soft 1018	Soft Cu	2400	1	104	0

*Notch Depth.

It is almost a foregone conclusion that severe cohesion would have occurred, at least between the two soft copper specimens, had some effective cleaning method been used. In fact this type of specimen looks very promising for classification of material pairs according to their sticking tendencies.

DISCUSSION

It has been found that pieces of soft copper tend to stick together at room temperature after being wire brushed in vacuum and slightly deformed in compression. Pieces of mild steel do not stick together under these conditions. Without wire brushing, pieces of soft copper do not stick to each other or to harder metals in vacuum even though severely deformed, by application of large forces to crossed chisel edges.

The major variables which determine the force required to separate two pieces of metal joined by a compressive load are hardness, cleanliness, deformation, temperature and time. It is assumed that maximum adhesion would occur if the surface could be made perfectly flat and clean. Whether this maximum would then represent perfect bonding regardless of temperature, hardness or time in contact is not known.

Cohesive stresses representing large fractions of the compressive yield stress were obtained on soft copper and soft steel in a previous program even at room temperature. However, these were obtained

by fracturing and rejoining with compressive strain at least equal to tensile strain, (sufficient to re-establish the original diameter) which was often quite a large strain. Furthermore, each part of a grain on the one surface was oriented at least approximately the same as the part left on the other surface and since grain orientation tends to increase cohesion, less cohesion might be expected with prepared flat surfaces under small amounts of compressive deformation than was obtained by the fracture-rejoin technique even if surfaces as clean as fractured surfaces can be prepared.

Bowden and Rowe (5) have developed formulae for predicting degree of cohesion from hardness and modulus of elasticity and relating it to friction coefficient. These formulae predict certain maximum limiting cohesion coefficient values for clean surfaces tested at temperatures low compared to those required for appreciable stress relief or diffusion in the time involved. If these are indeed fundamental limits, then little or no cohesion can be expected between flat faces of the harder specimens under observation; though appreciable sticking of hard specimens to soft copper might have been expected. Though these formulae agree with the maximum cohesion values observed by Bowden and Rowe at room temperature after cleaning in vacuum at temperatures so high as to cause observable specimen evaporation, there is still some question as to whether they apply to perfectly clean surfaces, particularly when they are quite smooth. Clean surfaces can be prepared by heating in vacuum but though clean while they are hot, they cannot be clean after cooling unless the integrated exposure during cooling is less than that required for the formation

of a small fraction of a monolayer of impinging gas, where the integrated exposure is defined as the area under a curve whose abscissa is the product of the pressure and the sticking coefficient (function of temperature) and the ordinate is time. It is extremely difficult to accomplish this or to demonstrate that it has been accomplished. For example, the careful work of Bowden and Rowe cited above did not even approach this condition.

Although metal surfaces in space applications could conceivably become perfectly clean due to abrasion, proton bombardment, etc., while in space, mating surfaces will never be perfectly flat or fit perfectly together. Furthermore, large compressive deformation of mating surfaces would seldom be encountered. Since the object of this work is to classify metals according to adhesive tendencies under practical conditions, and not to develop cold welding techniques, neither extremely smooth surfaces nor large deformation are of particular interest. However, the indications are that unless a more effective method of surface cleaning can be developed, more deformation than is now being applied will have to be used in order to obtain adhesive forces large enough to accomplish the desired classification of materials. Sliding or fretting at velocities low enough to cause little heating of the bulk materials are believed to have the same general effect as deformation applied by simple compression, i.e. to increase contact area and reduce stress gradients at the interface. Therefore, in the absence of an oxide film, sliding and deformation may be regarded as intensifiers which if applied in a controlled manner could be used to bring adhesive forces up to

conveniently measurable values which will still reflect the initial sticking tendencies of interest. If it were not for the small initial sticking tendency, sliding or fretting would have no effect and galling could not occur.

However, application of controlled amounts of sliding and deformation is difficult. Data obtained in air after twisting through a fixed angle under partial normal load is so scattered that it can be analyzed only on a statistical basis (6). It is believed that this scatter (in air) is due partly to the presence of the oxide film. However, slow sliding between clean surfaces in vacuum is just another form of deformation and it is believed that the combination of sliding and deformation which occurs between two 90° chisel edges of right angles can be more accurately controlled and permits collection of more reproducible data then the twisting technique. When the edges are of the same hardness, little sliding but considerable deformation occurs. For edges of different hardness considerable sliding occurs but deformation is less and occurs only on the softer specimen. Although the use of such specimens (90° crossed edges) requires the use of a more flexible wire brush than is now used if this method of cleaning is to be further investigated, it appears more likely in the light of the latest results that wire brushing will be dispensed with in favor of ion bombardment. The 90° chisel faces will be even more suitable than flat faces for this method since sputtering is considerably greater at 45° than at 90° (7).

It appears from the bake-out operations that the apparatus is suitable for testing specimens heated by radiation to at least 200°C. Since space vehicles or moon shelter parts might actually attain such a temperature, it is proposed that some of the materials be tested at 200°C as well as room temperature. Although the apparatus was designed for room temperature testing, internally mounted quartz lamps now used for bake-out appear to work so well that testing up to at least 200°C is considered practical.

CONCLUSIONS

Soft copper has no tendency to adhere to itself or to steel, titanium or Cu-Be alloy at 10^{-9} torr and room temperature after exposure to a pressure of 10^{-6} torr at 250°C, even when severely deformed in compression.

Wire brushing at 10^{-9} torr after heating to 250°C at 10^{-6} torr can cause at least 6% cohesion between flat faces of soft copper but not of soft steel at room temperature when slightly deformed in compression.

Wire brushing of soft copper at 10^{-9} torr after heating to 250°C at 10^{-6} torr does not cause it to adhere to unbrushed steel, titanium or Cu-Be alloy, at room temperature after slight deformation in compression.

No cohesion occurs between specimens of soft steel or of soft titanium when severely deformed in compression at 10^{-9} torr after exposure at 10^{-6} torr at 250°C.

Much less cohesion occurs between pieces of soft copper after wire brushing in vacuum than after fracturing and re-joining in vacuum.

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APPENDIX A

CLEANING A SURFACE BY ION BOMBARDMENT

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Ion bombardment cleaning of surfaces can be accomplished by the use of an ion gun which is used in a high vacuum chamber, or by initiating an electrical discharge between the surface and an adjacent positive electrode in a system at 10^{-2} - 10^{-1} torr. The latter technique has several disadvantages: First, the maintenance of a relatively high pressure requires valves to isolate the diffusion pumps. Second, measurements of scattering or emission effects is impossible. Third, material is often sputtered from other parts of the system causing contamination. Fourth, maintenance of the purity of the gas in the system is difficult.

Therefore, it is preferable to use an ion gun which can direct a beam of ions at the specimen surface only, thus permitting the system to operate at low pressures even during the cleaning period.

The ion gun must have a pressure of 10^{-2} torr within the gun cavity to maintain the discharge. An ion beam is emitted through an orifice and is directed to the target specimen. Some idea of the pressure which can be maintained in the system can be obtained by comparing the amount of gas typically effusing from the gun with the pumping speed of the system. A gun with an internal pressure of 10^{-2} torr and an orifice of 0.010-inch diameter would effuse approximately 6×10^{-5} torr liters per second of air. If we assume a net pumping speed of 1000 liters per second at the exit from the test system

then the system pressure would be 6 x 10^{-8} torr with the ion gun operating. The advantage of the ion gun over a local discharge to the specimen in a large chamber is that the ratio of the impingement rate of the desired species (Xe^+, for example) to that of undesired species (O_2, O^+, CO, CO^+, CO^{++}, etc.) is larger and more precisely known. Furthermore, the energy and species (Xe^+ or Xe^{++}) of the impinging inert gas ions are more controllable. The number of impinging nonionized inert gas atoms may be greater or less than in a local discharge but this is of little consequence.

It is important to control the energy of the impinging ions since ions even of the inert gases penetrate into the metal lattice if they impinge with too great an energy. This is shown by curves of "sticking coefficient" versus voltage, obtained by measuring the amount of inert gas given off on subsequent heating in vacuum. Apparently, the ions actually form substitutional solid solutions with metals and diffuse out on subsequent heating according to the usual diffusion laws. However, the sticking coefficient curves indicate zero sticking (penetration) below a critical voltage for each type of ion. For example, argon ions penetrate into tungsten above 150 volts but Xe ions require 200 volts.

To remove inert gases from metals requires relatively high temperatures since the activation energies of diffusion appear to be comparable to that for self-diffusion of the metal itself. The apparatus being used is not suitable for high temperature specimen outgassing, and since the magnitude of the effect of inert gas ions in solid solution on hardness and strain hardening coefficient (which affect cohesion) is not known, it seems advisable to stay below the

critical sticking coefficient voltages if possible. Data in the literature indicate that good "cleaning" (ratio of sputtered atoms or ions to impinging ions) is possible below these voltages but there is some question as to how efficiently the ion guns can be made to work at such low voltage. Obviously, the heavier the impinging ion the better, since a larger percentage of the energy is given up near the surface and higher voltage can be used without penetration. Therefore, xenon should be better than argon. Actually, it is almost impossible to clean metal surfaces in vacuum at low temperature without changing the physical characteristics of the metal surface somewhat. Abrasion, even by a sharp wire brush, undoubtedly work hardens the surface to some extent.

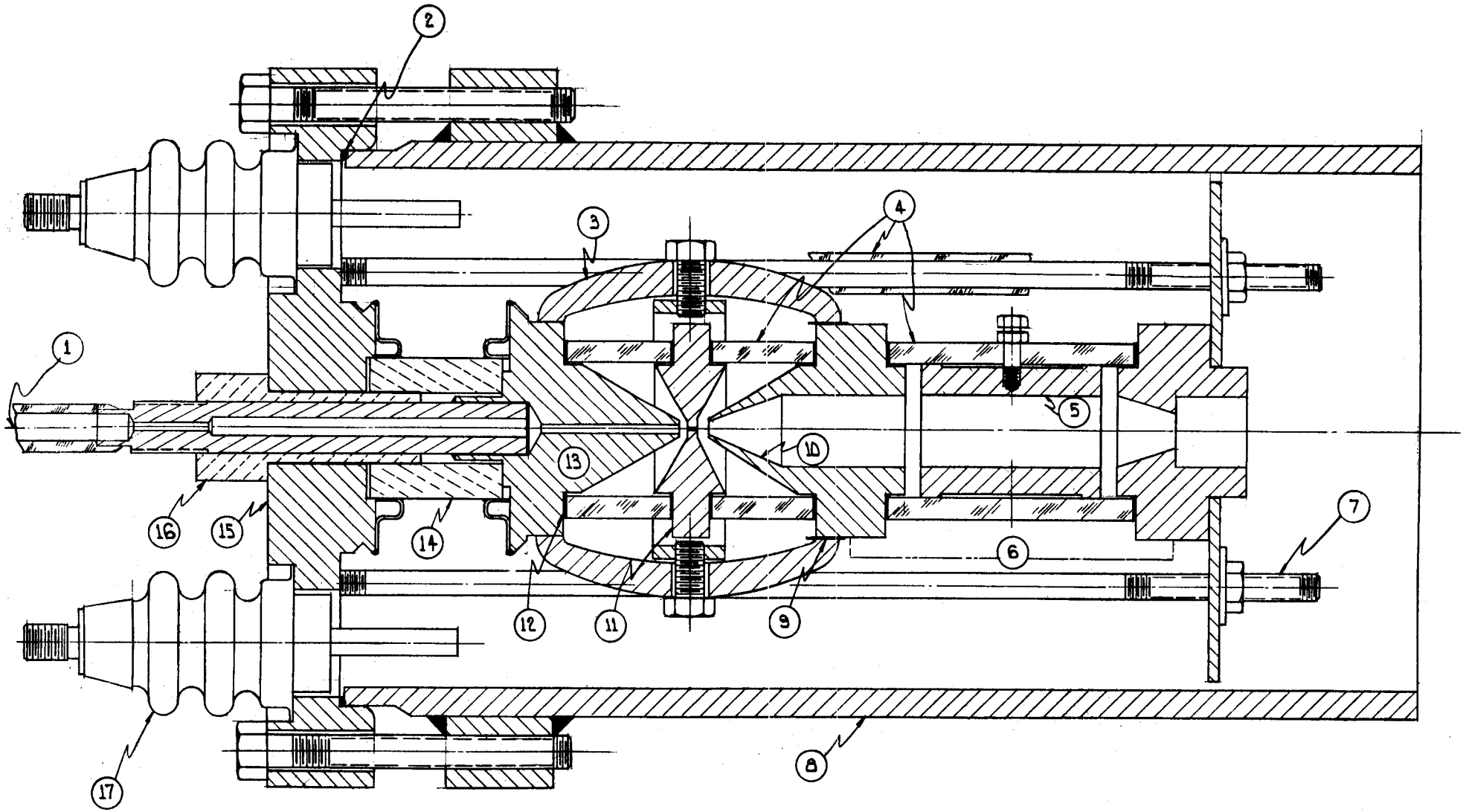
There is considerable interest in the effect of proton bombardment since the proton flux in space and on the moon is apparently sufficient to remove surface oxides at least from some metals in reasonable periods of time. In fact, proton bombardment is the only mechanism aside from abrasion by which most metals could lose their oxide films in reasonable periods of time in space except at high temperatures. Therefore, although proton bombardment was not included in this program it is hoped that the ion guns will prove suitable for operation with hydrogen as well as with xenon, and for operation at the 10 kv or so required for proton flux space simulation. The flux density of protons in space is low enough to be easily exceeded in the laboratory.

ION GUN DESIGN

Design of the ion guns was preceeded by considerable study of the literature. None of the commercially available units were suitable for direct attachment to the apparatus. Therefore, a specially designed gun was fabricated. The design is shown schematically in Fig. 1A.

Pure xenon gas is bled in at the rate required to maintain a pressure of 10^{-2} torr to 10^{-1} torr, on the inlet side of the 0.010-inch diameter orifice in the aluminum orifice plate. A discharge is to be established by applying a suitable voltage at radio frequencies, if necessary, between the orifice plate and the conical pole piece through which the gas enters. An intense magnetic field is established between this conical pole piece and the conical pole piece on the other side of the aluminum orifice plate, by means of the permanent magnets clamped to the hexagonal faces of these pole pieces which are composed of an iron-cobalt alloy with high saturation flux density. Mica is placed under one end of the magnets for electrical insulation. The second pole piece also serves as the ion extractor which may require as much as 30 kv for efficient extraction. The ions then pass through an Einzel lens with its center section in three segments to permit beam deflection as well as focusing.

The whole assembly including the magnets (which are polished) is located within the vacuum system, the discharge chamber being isolated by aluminum gaskets between the flat ground ends of the tubular glass insulators and machined faces on the pole piece and



ION GUN

Figure 1A

LEGEND FOR FIGURE 1A - ION GUN

1. Gas Inlet
2. Gold "O" Ring
3. Magnets (six)
4. Glass Tubes
5. Aluminum Deflectors (Three Sectors)
6. Einzel Lens
7. Tie Rods
8. Stainless Steel Pipe (4" diameter)
9. Mica
10. Ion Extractor (and pole piece)
11. Aluminum Orifice Plate
12. Aluminum Foil
13. Pole Piece (Fe-Co) (Hexagonal Faces)
14. Ceramic Insulator
15. Stainless Steel Plate
16. Boron Nitride Bushing
17. Ceramic Insulators (six)

orifice plate. The assembly is mounted on a thick stainless steel plate which fits on the end of the 4-inch diameter vacuum chamber side arm. A gold "O" ring .030-inch in diameter will be used for the vacuum seal at this point. All electrical connections are brought out through ceramic-to-metal sealed insulators, mounted in the plate. Construction is such as to permit baking out at temperatures usually used for Pyrex glass.

These guns are of the type which requires the establishment of a small volume of intensely ionized gas on one side of a small orifice and the extraction of ions into a relatively good vacuum on the other side of the orifice by a conical high voltage electrode. An axial magnetic field is provided to help keep the zone of ionization or plasma near the center. Some guns of this type use hot cathodes, and some use R.F. to maintain the plasma on the high pressure (20 to 100 microns) side of the orifice. The amount of ion current obtainable from such guns depends largely on the degree of ionization of the gas just in front of the orifice. To obtain currents equivalent to complete ionization requires extremely intense arc type discharges and the attendant use of water cooling, refractory materials, etc.

Since a flow of only .177 torr liters per second represents one ampere of singly charged ions and since the flow of xenon involved (.010" orifice at 3×10^{-2} torr) is about 3×10^{-4} torr liters per second, complete ionization and extraction would correspond to about 1.0 milli-ampere.

Since only 10^{16} impingements should be required for cleaning and one ampere corresponds to 6.24×10^{18} impingement per second, a current of about 1 micro-ampere should be sufficient for cleaning in 1000 seconds. This means that at least 0.1% of the gas passing through the orifice must be ionized and guided to the specimen. It was felt that this should be possible using a low power R.F. or D.C. glow discharge at 10^{-1} to 10^{-2} torr, concentrated by permanent magnets.

The holes in the orifice plates are .010" diameter and .005" to .010" long. The magnets were polished and then remagnetized. Figure 2A shows one of the guns assembled except for wiring. The base plates were bolted in place against the gold "O" rings for the pumpdown and bake-out and found to be vacuum tight. A low power high voltage power supply was procured for ion extraction. Several other power supplies were also required. After installing xenon gas bottles, special valves for gas flow control and the necessary wiring and instrumentation, the appearance of the apparatus was as shown in Figure 3A.

Two electron collecting devices were also built. These are small metal cylinders with a conical hole, an anode and a grid to be mounted at an angle to the specimen face while it is bombarded with xenon ions. On the basis of a literature survey it is anticipated that the relative degree of cleanliness of the specimen surfaces can be reduced by measuring the relative number of electrons produced per xenon ion impingement.

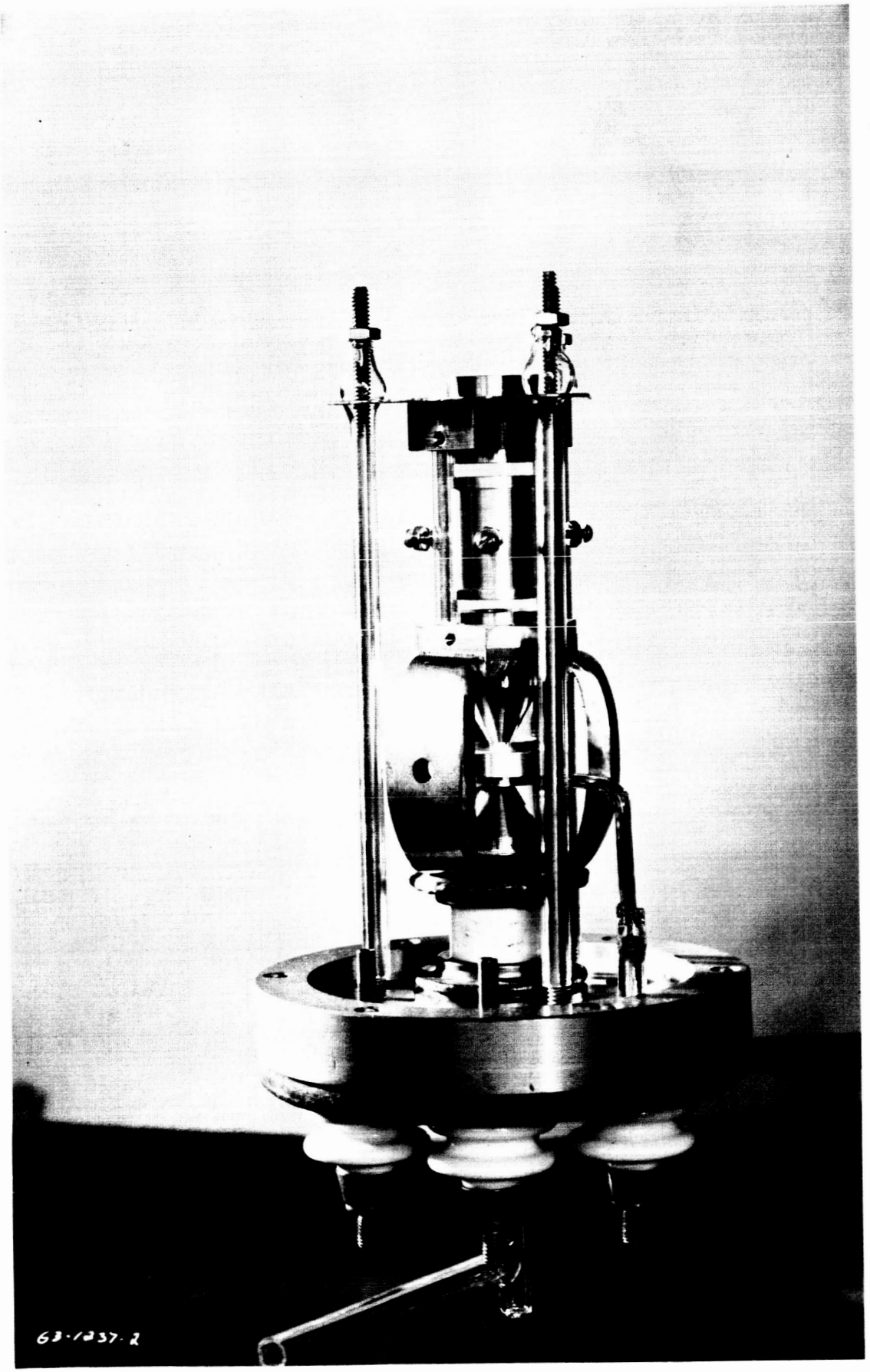


Figure 2A - Ion Gun

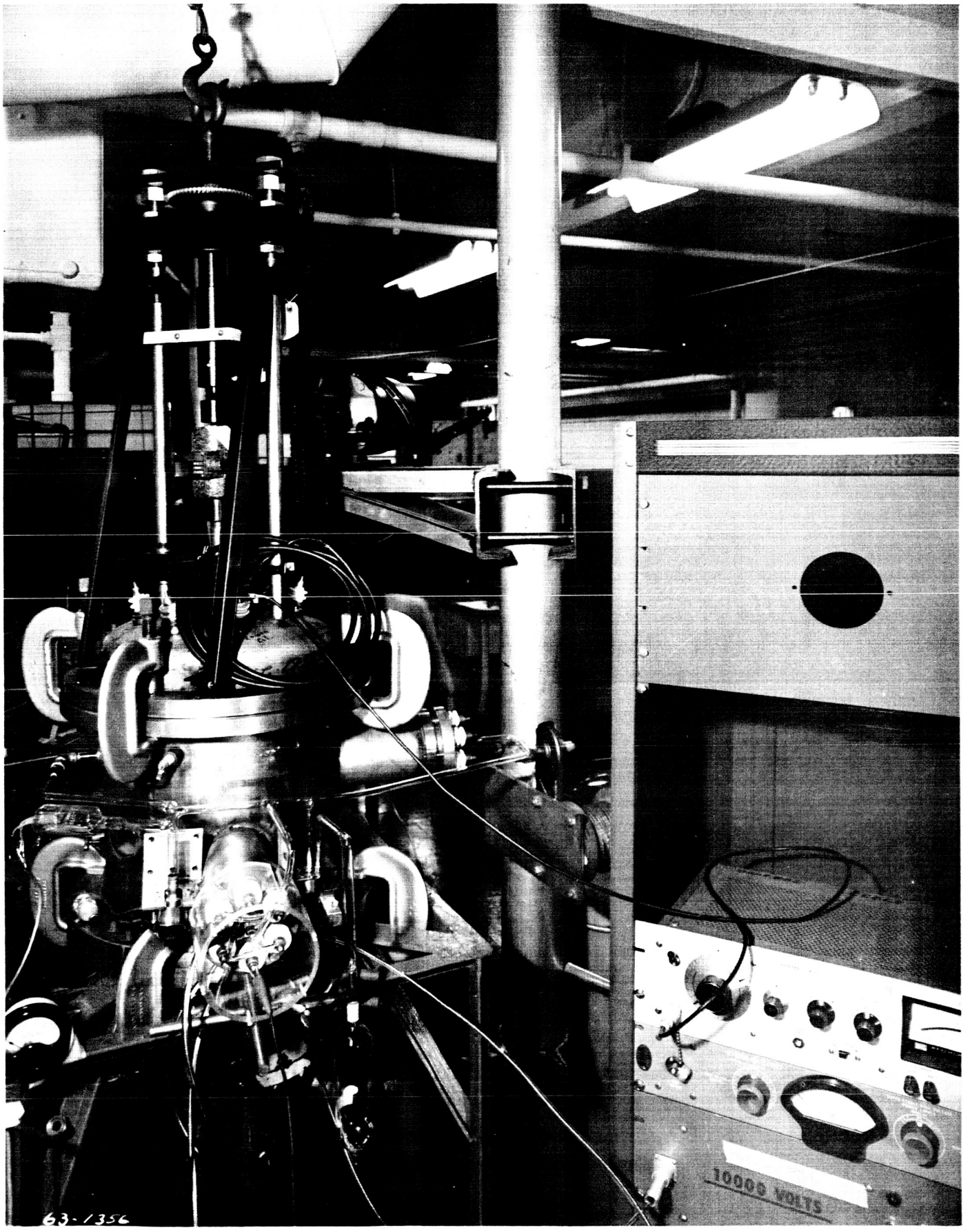


Figure 3A - Cohesion Apparatus Complete with Ion Guns

The apparatus was evacuated and various D.C. voltages applied to the elements of the ion guns while varying the pressure of xenon. Plates coated with a phosphor and grounded through a microammeter were used as targets instead of specimens.

The major problem with the ion guns was the maintenance of a sufficient ion current between the orifice plate and the gas inlet cone to furnish the required reservoir of positive ions. The xenon inlet pressure was varied from 25 microns to 300 microns, corresponding to 5×10^{-6} torr and 9×10^{-5} torr vacuum chamber pressure. The orifice plate was kept at +200 volts above ground, since it determines the final ion velocity and higher voltages are reported to cause xenon occlusion. At first, discharge occurred through the external glass tubing to the metering valves. After these were insulated 400 μa was obtained in the reservoir at 160 to 190 microns Hg xenon pressure using 500 volts D.C. With 2 kv on the ion extractors, only $.4 \times 10^{-10}$ amps ion current was thus obtained. At 3 kv a small negative current was obtained at the target and at 5 kv an erratic discharge occurred. Application of a Tesla coil to either the orifice plate or the inlet cone doubled the ion current in the reservoir but interfered with the microammeter reading to the target.

The following measures were then taken in an effort to establish an ion beam. The magnets were removed from one gun (two are being tested) in order to simplify internal insulation problems and reduce the apparent resistance of the gas in front of the orifice plate. The

other gun was fitted with a tungsten filament heater inside the inlet cone near the orifice. An RF oscillator (150 watt) was obtained to assist in generating ions and a higher voltage supply obtained for the ion extractor. A variety of circuits were then tried in quick succession but it soon became apparent that visual observation of the shape and location of the plasma was absolutely essential and that the necessary visibility would have to be provided before further attempts would be worthwhile. It is believed that a simple R.F. discharge between the inlet cone and the orifice plate, as originally planned, may suffice if windows for viewing the discharge are provided. The next step in perfecting the guns is to mount one in a glass tube on a separate system and run tests using argon.